Direct Preparation of Particles from Liquid Suspension for Rapid Identification and Screening

There are several complicated multistep procedures that are commonly used for the preparation of particles for analysis by scanning electron microscopy. One procedure involves the suspension of particles in a liquid and pipeting an aliquot of the liquid onto a carbon planchet for evaporation, however the receding liquid often causes agglomeration and clumping of the particles. A second involves filtering a liquid suspension through a commercial filter material and then removing the filter material so the particles lay down on a carbon planchet for imaging and microanalysis in a scanning electron microscope (SEM). This work draws on the strengths of both of these methods.

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In the NIST-developed sample preparation procedure for SEM analysis, particles suspended in liquid are filtered onto the thinned carbon planchet on a vacuum filtration apparatus. The particles are separated from each other and on a substrate that can be imaged directly in the SEM. This preparation procedure is based on the observation that carbon disks of the type commonly used for electron microscopy are porous and can be used as liquid filters. In addition if the disk is thinned to between 1 mm and 2 mm in thickness, the filtration is quick enough that particle settling and aggregation is minimized. We evaluated three grades of commercially available carbon graphite rods as potential sources for the planchet filter substrates. The rods were cut into discs and evaluated as cut from the rod by diamond saw and in a diamond polished final state. The different grades and finishes were evaluated for filtration time (Table 1). However, filtration time alone is not sufficient to evaluate these planchets because if all the particles are lost, then it is not a useable technique. The grades and finishes were also evaluated for particle retention, calculated as effective pore size and removal efficiency (Table 2). The *Effective Pore Size* shown in the table is defined as the smallest particle removed with a minimum of 90 % efficiency. Finally, the resulting planchet filters were tested with two real world cases. The first example was the filtration of medium test dust particles from hydraulic fluid (NIST SRM 2806). The second demonstration involved the filtration onto a gold-coated planchet filter of suspended materials from a natural water source.

This effort resulted in the development of an effective low-cost mounting procedure for particle analysis in scanning electron beam instruments. We recommend high-quality isomolded polished planchets for filtration and direct observation of particles by electron microscopy.

Table 1. ***Filtration Time (minutes)***

Carbon Planchet Sample	DI H_2O Flush n=9	Fly Ash n=3	SRM 2806 n=2	Sligo Creek n=2
High-Quality Polished	8.9±3.7	49.3 ± 16.3	21	96 *
High-Quality Unpolished	1.3 ±1.4	3.5 ± 3.4	10	----
Med.-Quality Polished	3.7± 1.6	66.8 ±27.5	----	----
Med.-Quality Unpolished	8.2± 5.3	22.1 ± 6.1	----	----
Low-Quality Polished	----	0.12 ± 0.01	----	----
Low-Quality Unpolished	----	0.06 ± 0.02	----	----
Millipore Filter	0.28 ± 0.02	0.05 ± 0.06	----	----

- * Gold coated
- Volume of DI flush and fly ash experiments was 15 mL for the medium quality and high quality samples and 60 mL for the low quality samples
- Volume of NIST SRM 2806 and Sligo Creek water was 10 mL
- Numbers reported are average ± standard deviation
- n is the number of replicates

Table 2.

Carbon Planchet Sample	Effective Pore Size (µm)	Removal Efficiency (%)	Standard Deviation (%)
High-Quality Polished	2.5	96	28
High-Quality Unpolished	4.5	93	47
Med.-Quality Polished	2.5	92	28
Med.-Quality Unpolished	2.5	92	59
Low-Quality Polished	34.5	91	25
Low-Quality Unpolished	35.5	91	82

Impact: The growing interest in micro/nano particle technology in many industry sectors makes this an economic alternative to the more time- and labor-intensive methods currently in use for the preparation of particle SEM samples from liquid suspensions.

Future Plans: We are currently investigating a sample preparation method for the dispersal of dry powders onto analysis substrates using camphor and naphthalene.

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